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(71)Applicant : FUJI XEROX CO LTD
(72)Inventor : YAMASHITA YOSHIRO
INOUE HIROSHI
HASHIMOTO TAKESHI

## (54) INK FOR INK JET RECORDING AND INK JET RECORDING

(57)Abstract:
PROBLEM TO BE SOLVED: To obtain the subject ink rapidly drying on a regular paper, capable of providing excellent print quality, water-resistance/abrasion resistance and long term preservation stability, and hardly causing plugging or the like by including water, a pigment self-dispersable in the water, a water-soluble organic solvent and a specific water-soluble organic compound in a specified amount.
SOLUTION: The objective ink comprises water, a pigment self-dispersible in the water, preferably having 15-100 nm number average dispersing particle diameter and ≤3.5 dispersion particle size distribution (e.g. carbon black such as Mitsubisi No.25), a water-soluble organic solvent and 3.0-15.0 wt.% water-soluble organic compound having ≤12 S.P. value and <40 mN/m surface tension {preferably, a compound of the formula R-O-XnH [R is a 1-8C alkyl or the like; X is oxyethylene or the like; (n) is 1-8], having <400 molecular weight and ≥8 S.P. value}.

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## CLAIMS

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[Claim(s)]
[Claim 1] surface tension [ in / 25 degrees C / value / S.P. / on the ink for ink-jet record which contains the pigment and water-soluble organic solvent in which self-distribution is possible in water and water, and / in 12 or less ] -- the water-soluble organic compound of less than 40 mN/m -- the total weight of ink -- being based -- 3.0wt% - 15.0wt% -- the ink for ink-jet record characterized by containing
[Claim 2] Ink for ink-jet record according to claim 1 in which the drying time at the time of usually printing the solid picture of 100% of rates of picture area in the paper is characterized by being less than 5 seconds.
[Claim 3] Ink for ink-jet record according to claim 1 in which the diameter of number-average part granule application of a pigment in which self-distribution in water is possible is characterized by being 15nm - 100nm.
[Claim 4] Ink for ink-jet record according to claim 1 in which the distributed particle size distribution of a pigment in which self-distribution in water is possible are characterized by being 3.5 or less.
[Claim 5] Ink for ink-jet record according to claim 1 in which the particle number which has a particle diameter exceeding 0.5 micrometers contained in ink is characterized by being three or less 6x1010 pieces / dm.
[Claim 6] Ink for ink-jet record according to claim 1 in which molecular weight of a water-soluble organic compound is characterized by being less than 400.
[Claim 7] Ink for ink-jet record according to claim 1 in which the S.P. value of a water-soluble organic compound is characterized by being eight or more.
[Claim 8] Ink for ink-jet record according to claim 1 characterized by a water-soluble organic compound being a compound shown by R-O-XnH (the functional group chosen from the group which consists of the alkyl, the alkenyl, the alkynyl, the phenyl, alkylphenyl, and cycloalkyl of R:C1-C8, X:oxyethylene or oxypropylene, integer of n=1-8).
[Claim 9] Ink for ink-jet record according to claim 1 in which surface tension of ink is characterized by being 20 mN/m - 40 mN/m.
[Claim 10] Ink for ink-jet record according to claim 1 in which viscosity of ink is characterized by being 1.8mPas - 4.0mPas.
[Claim 11] Ink for ink-jet record according to claim 1 in which the conductivity of ink is characterized by being 0.03 S/m - 0.4 S/m.
[Claim 12] The manufacture method of the ink for ink-jet record characterized by including the process which distributes underwater the pigment in which self-distribution in water is possible using an ultrasonic homogenizer or a high-pressure homogenizer, and the process with which surface tension [ in / 25 degrees C / value / S.P. / the dispersed pigment, water, the water-soluble organic solvent, and / in 12 or less ] mixes the water-soluble organic compound of less than 40 mN/m.
[Claim 13] The manufacture method of the ink for ink-jet record according to claim 12 characterized by including the big and rough particle removal process by centrifugal separation.
[Claim 14] Claims 1-11 are the ink-jet record methods characterized by making the ink of a

publication breathe out from an orifice according to a record signal, and recording on a recorded material either.
[Claim 15] The ink-jet record method according to claim 14 characterized by making heat energy act on ink and making ink breathe out.
[Claim 16] The ink-jet record method according to claim 14 characterized by impressing two or more pulses and making the drop of one ink form and breathe out.
[Claim 17] The ink-jet record method according to claim 14 characterized by including the process which mixes the breathed-out ink and a binder on a recorded material.
[Claim 18] The ink-jet record method according to claim 14 that discharge quantity per 1 drops is characterized by 1 or more ngs being 20 or less ng.

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[Translation done.]

## DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[The technical field to which invention belongs] this invention relates to the ink for ink-jet record used for the recording devices (a printer, a copying machine, facsimile, a word processor, plotter, etc.) of an ink-jet method, its manufacture method, and the ink-jet record method using it.
[0002]
[Description of the Prior Art] A liquid or melting dry ink is breathed out from a nozzle, a slit, a porosity film, etc., the so-called recording device of the ink-jet method which records on recorded materials, such as paper, cloth, and a film, is small, and since it has advantages, such as cheapness and calm nature, various examination is performed. In recent years, not only the black monochrome printer by which the so-called good qualities of printed character, such as a report form and a copy paper, are obtained but many products which can perform full color record are usually marketed in the paper, and it came to occupy a big position in the field of the recording device.
[0003] The ink used by the ink-jet recording device usually mainly consists of a solvent, color material, and an additive. About the ink for ink-jet record, the blot in (1) paper, high resolution without a fogging, and a picture uniform at high concentration are acquired, (2) The blinding at the nose of cam of a nozzle by ink dryness does not occur, but there is demand characteristics, like that regurgitation responsibility and regurgitation stability are always good, that the robustness of that the drying property of the ink in the paper is good and (3) (4) picture is good, and (5) mothball stability is good.
[0004] Various meanses are provided as a remedy for filling such demand characteristics. For example, the drying property of the ink in the paper of the above (3) is important in order to raise the speed of a recording device or to prevent the color mixture blot at the time of performing color printing, the high solvent of permeability or volatility is used, or the improvement by adding a surfactant etc. has been made conventionally.
[0005] Moreover, about the robustness of the picture of the above (4), many ink which has improved water resistance is indicated by using a pigment instead of a color as a color material (JP,56-147869,A, JP,2-255875,A, U.S. Pat. No. 5085698 specification, etc.). However, although water resistance improves when pigment ink is used, there is a fault that scratch-proof nature is inferior compared with color ink. Furthermore, when pigment ink is used, there is also a problem of becoming what is generally inferior compared with color ink about the mothball stability of the above (5).
[0006] The method of oxidizing carbon black is indicated by the method to which a carbon black front face is made to carry out the polymerization of the water-soluble monomer etc., and JP,8-3498,A at the method of carrying out azo coupling of the substituent which contains a water solubilization machine in carbon black to for example, a U.S. Pat. No. 5571311 specification as a method of solving the problem at the time of using a pigment, and JP,8-81646,A. However, mothball stability is good, a drying property in the paper is usually quick, a good quality of printed character is obtained, and the ink for ink-jet record which was moreover excellent in water resistance / scratch-proof nature is not yet found out.

[0007]
[Problem(s) to be Solved by the Invention] Therefore, the technical problem of such a Prior art is solved, mothball stability is good, a drying property in the paper is usually quick, a good quality of printed character is obtained, and the purpose of this invention aims at offering the ink which was moreover excellent in water resistance / scratch-proof nature.
[0008]
[Means for Solving the Problem] the water-soluble organic compound whose surface tension [ in / 25 degrees C / on the ink for ink-jet record containing the pigment which this invention persons can self-distribute in water, the water-soluble organic solvent, and water, and / in an S.P. value ] is less than 40 mN/m or less in 12 -- the total weight of ink -- being based -- 3.0 - 15.0wt% -- by containing, it found out that the above-mentioned purpose could be attained and this invention was completed
[0009] Namely, this invention is set in the ink for ink-jet record which contains the pigment and water-soluble organic solvent in which self-distribution is possible in water and water. S. Surface tension [ in / 25 degrees C / in P. value ] or less by 12 the water-soluble organic compound of less than 40 mN/m the total weight of ink -- being based -- 3.0 - 15.0wt% -- the pigment in which self-distribution in the ink for ink-jet record and water which are characterized by containing is possible with the process underwater distributed using an ultrasonic homogenizer or a high-pressure homogenizer The manufacture method of the ink for ink-jet record characterized by the dispersed pigment, water, the water-soluble organic solvent, and an S.P. value including the process with which the surface tension in 25 degrees C mixes the water-soluble organic compound of less than 40 mN/m or less by 12, And the aforementioned ink is made to breathe out from an orifice according to a record signal, and it consists of the ink-jet record method characterized by recording on a recorded material.
[0010] Hereafter, this invention is explained in detail. Even if "the pigment in which self-distribution in water is possible" used in this invention does not have existence of a dispersant in the front face including many solubilization machines to water, it is a pigment distributed stably. In order to be "the pigment in which self-distribution in water is possible" of this invention Distributed equipments, such as an ultrasonic homogenizer, a nano mizer, a Micro fluidizer, and a ball mill, are used. When the pigment concentration of a supernatant is measured after having made water distribute a pigment, without using a dispersant so that it may become concentration (water 95wt%/pigment 5wt%), measuring initial pigment concentration and leaving dispersion liquid with a glass bottle after that on the 1st, the pigment concentration should just be 98% or more of initial concentration.
[0011] "The pigment in which self-distribution in water is possible" of this invention can be manufactured by performing surface-treatment processing of acid-base processing, coupling-agent processing, polymer graft processing, plasma treatment, or oxidization/reduction processing to the usual pigment. By performing such surface treatment, more solubilization machines to water than the usual pigment come to be included, and self-distribution is attained. As a usual pigment to which surface-treatment processing is performed Raven 5250 Raven 3500 Raven 5750 Raven 1080 Regal 330R, Mogul L, Monarch 1000 Color Black FW2, Black Pearl L, Printex V, Special Bk Although the carbon black of 4A and Mitsubishi No.25 grade can be mentioned as a desirable example C. I. Pigment Blue1 and C.I. Pigment Blue 3 and C.I. Pigment Blue 15 C.I. Pigment Blue 15-3, C.I. Pigment Blue 16 C.I. Pigment Blue The cyano pigment of the 60th grade, C. I. Pigment Red 5 and C.I. Pigment Red12 and C.I. Pigment Red 48 and C.I. Pigment Red 112 and C.I. Pigment Red 122 C.I. Pigment Red 146 C.I. Pigment Red 168 C.I. Pigment Red The Magenta pigment of the 202nd grade, C. I. Pigment Yellow 1 C.I. Pigment Yellow 2, C.I.Pigment Yellow 3 C.I. Pigment Yellow 13 C.I. Pigment Yellow 16 C.I. Pigment Yellow 73 C.I. Pigment Yellow 83 C.I. Pigment Yellow 98 C.I. Pigment Yellow 114 C.I. Pigment Yellow 128 C.I. PigmentYellow The color pigment of the 154th grade It can use.
[0012] Moreover, "the pigment in which self-distribution in water is possible" of this invention can also use for commercial water the pigment in which self-distribution is possible as it is. As an example of the pigment of such marketing, they are Cab-o-jet -200, Cab-o-jet -300, IJX-55 (above, Cabot Corp. make), and Microjet. Black CW-1 (Orient chemistry company make) etc. is

mentioned.
[0013] Although the solubilization machines to the water contained in the pigment in which self-distribution in water is possible may be Nonion nature, cation nature, and anionic any, a sulfonic acid, a carboxylic acid, a hydroxyl group, its phosphoric acid, etc. are mainly desirable. Although it can use also in the state of a free acid as it is in the case of a sulfonic acid, a carboxylic acid, and a phosphoric acid, it does not matter even if it forms the salt. When the salt is formed, as for the counter ion of an acid, generally, it is desirable that they are Li+, Na+, K+, NH4+, and an organic amine.
[0014] this invention -- setting -- the content of the pigment in which self-distribution in water is possible -- a total ink weight -- being based -- desirable -- 0.1 - 20wt% -- more -- desirable -- 0.5 - 15wt% It is 1 - 10wt% especially preferably. If the content of a pigment exceeds 20wt (s)%, it will become easy to produce the blinding in the nose of cam of a nozzle. Sufficient concentration will not be obtained if a content becomes less than [ 0.1wt% ].
[0015] Moreover, as for the diameter of number-average part granule application in the ink of the pigment in which self-distribution in water is possible, it is especially desirable the range of 15nm - 100nm and that it is the range of 15nm - 50nm. If the diameter of number-average part granule application is in this range, it will be hard to produce blinding and will become the thing excellent also in preservation stability. If the diameter of number-average part granule application is set to less than 15nm, probably because the surface area per particle unit volume will become large and the touch area between particles will become easy to become large in ink, ink viscosity becomes high and tends to produce blinding. On the contrary, if a mean particle diameter exceeds 100nm, distribution will tend to become unstable and the inclination which leads to condensation and sedimentation of a pigment will be seen.
[0016] Moreover, as for especially the distributed particle size distribution (ratio to the diameter of number-average part granule application of volume mean-dispersion particle size) of a pigment in which self-distribution in water is possible, it is [ 3.5 or less ] desirable that it is 2.5 or less. When distributed particle size distribution exceed 3.5, even if the diameter of number-average part granule application is in the above-mentioned range, there is an inclination which becomes easy to cause condensation and sedimentation by using some big particulate materials as a nucleus. "used in this invention -- S. -- P. value -- 12 or less -- surface tension -- water-soluble organic compound" of less than 40 mN/m -- the total weight of ink -- being based -- 3.0wt% - 15.0wt% -- the value of distributed particle size distribution can be made or less into 3.5 by containing
[0017] Furthermore, when using the ink-jet recording method characterized by making ink breathe out by operation of heat energy, in order to suppress the burn on a heater, it is desirable <=6x1010 piece / dm3, and to set especially to <=3x1010 piece / dm3 the particle-diameter >0.5micrometer particle number contained in ink. Particle-diameter > In order to make a 0.5-micrometer particle number into the above-mentioned range, in manufacture of ink, it is desirable to take in the big and rough particle removal process by centrifugal separation or filtration. If the particle-diameter >0.5micrometer particle number contained in ink exceeds 6x1010 pieces / dm3, it may become the cause of blinding or sedimentation of a pigment may arise during preservation, or the burn on a heater is accelerated and the fall of the amount of drops may arise. If a lot of surfactants are added in order to bring the drying time forward, although a particle number will tend to increase by the usual pigment-content powder system, it becomes possible to suppress a >0.5micrometer particle number by using the pigment in which self-distribution is possible.
[0018] An S.P. value (Solubility Parameter) is 12 or less thing, and, as for the water-soluble organic compound used in this invention, it is desirable especially that they are or less 12 eight or more things. S.P. The drying time will become long if a value exceeds 12. Moreover, if an S.P. value becomes less than eight, it will become easy for the solubility to water to fall. Moreover, the surface tension in 25 degrees C is the thing of less than 40 mN/m, and, as for the water-soluble organic compound used in this invention, it is desirable especially that it is 20 or more mN/m the thing of less than 40 mN/m. If surface tension becomes 40 or more mN/m, the wettability to a recorded material will become bad and neither a drying property nor abrasion

resistance will be improved. Moreover, since it gets wet too much to record material when it comes to less than 20 mN/m, it becomes easy to produce a blot and a strike-through. Moreover, less than 400 thing has [ the water-soluble organic compound used in this invention ] desirable molecular weight. If molecular weight becomes 400 or more, since the blinding nature of a nozzle will become bad, it is not desirable.

[0019] The compound which has the structure shown by R-O-XnH (the functional group chosen from the group which consists of the alkyl, ARUKENIRU, the alkynyl, the phenyl, alkylphenyl, and cycloalkyl of R:C1-C8, X:oxyethylene or oxypropylene, integer of n=1-8) from the point of pigment-content powder stability as a water-soluble organic compound which satisfies these requirements is desirable. Moreover, it is desirable in the above-mentioned formula that R is C3-C6 and n is especially the integer of 1-6. As an example of the compound which has the structure shown by the above-mentioned formula An ethylene glycol monobutyl ether, the ethylene glycol monopropyl ether, The diethylene-glycol monobutyl ether, the diethylene-glycol monochrome hexyl ether, The diethylene-glycol monopropyl ether, the dipropylene-glycol monopropyl ether, The dipropylene-glycol monobutyl ether, the propylene-glycol monobutyl ether, the propylene-glycol monopropyl ether, the triethylene-glycol monobutyl ether, a diethylene-glycol monochrome phenyl ether, etc. are mentioned. Also in these, the diethylene-glycol monobutyl ether, the diethylene-glycol monochrome hexyl ether, the propylene-glycol monobutyl ether, and the triethylene-glycol monobutyl ether are desirable.

[0020] such [ the ink for ink-jet record of this invention ] a water-soluble organic compound -- the total weight of ink -- being based -- 3.0wt% - 15.0wt% -- desirable -- 3.0wt(s)% - 10.0wt% -- it contains If a content becomes less than [ 3.0wt% ], the pass-through effect will seldom be discovered and a rate of drying will fall. On the contrary, if 15.0wt% is exceeded, the obstacle that become or the distributed stability of the pigment in which self-distribution is possible falls to water by too much osmosis that it is easy to produce a blot will arise.

[0021] The water-soluble organic solvent used in this invention Although it will not be limited especially if it is used in order to prevent evaporation of water in ink-jet ink For example, ethylene glycol, a diethylene glycol, a propylene glycol, A polyethylene glycol, a triethylene glycol, a glycerol, a trimethylol propane, Polyhydric alcohol, such as 1, 2, 6-hexane triol, 1,5-pentanediol, and a dipropylene glycol Saccharides, such as a glucose, a fructose, a galactose, a mannose, and a xylose, Thiodiethanol, 2-mercaptoethanol, the thioglycerol, Nitrogen-containing solvents, such as sulfur-containing solvents, such as a sulfolane and dimethyl sulfoxide, 2-pyrrolidone, a N-methyl-2-pyrrolidone, a cyclohexyl pyrrolidone, a monoethanolamine, a triethanolamine, and a diethanolamine, can be used. Also in these, ethylene glycol, a diethylene glycol, a propylene glycol, a glycerol, thiodiethanol, a sulfolane, 2-pyrrolidone, a N-methyl-2-pyrrolidone, etc. are desirable. Moreover, even if it uses these independently, they may mix and use two or more sorts. As for especially the content of the water-soluble organic solvent, based on the total weight of ink, it is desirable to consider as about 3 wt(s)% - 50wt% about 1 wt% - 60wt%. If 60wt (s)% is exceeded, ink viscosity will rise and **** stability and **** responsibility will fall. Moreover, when it comes to less than [ 1wt% ], suppressing [ of evaporation of water ] is easy to become inadequate, and it becomes easy to start blinding.

[0022] although especially the water used in this invention is not limited -- pure water -- desirable -- the total weight of ink -- being based -- 35wt% -- being contained -95wt is desirable If when it comes to less than [ 35wt% ] the distributed stability of the pigment in which self-distribution is possible gets worse in water, or ink viscosity may become high, **** stability may fall and 95wt% is exceeded, evaporation tends to increase at the nozzle edge and may produce blinding.

[0023] As a **** stabilizing agent, the ink for ink-jet record of this invention can mix independent or two sorts or more, and can contain a urea, thiourea, an ethylene urea, an ethylenetiourea, methylurea, a dimethyl urea, a methylthio urea, dimethyl thiourea, etc. As for especially the content of these **** stabilizing agents, it is desirable to the weight of ink to consider as about 1 wt% - 10wt% about 0.5 wt(s)% - 15wt%.

[0024] Moreover, the ink for ink-jet record of this invention As a surfactant, a Nonion nature activator, for example, polyoxyethylene alkyl phenyl ether, Polyoxyethylene alkyl ether,

polyoxyethylene fatty acid ester, A sorbitan fatty acid ester, polyoxyethylene sorbitan fatty acid ester, Polyoxyethylene glycerine fatty acid ester, polyglyceryl fatty acid ester, Polyoxyethylene sorbitol fatty acid ester, a polyoxyethylene sterol, The polyoxyethylene polyoxypropylene ether, polyoxyethylene fatty acid amide, A polyoxyethylene polyoxypropylene block copolymer, a tetramethyl crepe-de-Chine diol, Anionic surface active agents, such as a tetramethyl crepe-de-Chine diol ethyleneoxide addition product, For example, alkylbenzene sulfonates, an alkylphenyl sulfonate, Alkylnaphthalenesulfonate, a higher-fatty-acid salt, the sulfate salt of higher-fatty-acid ester, The sulfonate of higher-fatty-acid ester, the sulfate salt of the higher-alcohol ether, and a sulfonate, The formalin condensate of a high-class alkyl sulfo succinic-acid salt and a naphthalene sulfonate, A polystyrene sulfonate salt, a polyacrylate, polyoxyethylene-alkyl-ether phosphate, The others which are an alkyl ether carboxylate, an alkyl sulfate, an acrylic-acid-acrylic-ester copolymer, etc., Silicone system surfactants and perfluoroalkyl carboxylates, such as a polysiloxane polyoxyethylene addition product, Biosurfactants, such as fluorine system surfactants, such as a perfluoroalkyl sulfonate and oxyethylene perfluoro alkyl ether, a SUPIKURISU pole acid, and a rhamnolipid, a lysolecithin, etc. can be contained. Independent or two sorts or more can be mixed and used for these surfactants.
[0025] As for HLB of a surfactant, it is desirable that it is the range of 5-25 in consideration of pigment-content powder stability. As for especially the addition of a surfactant, based on the total weight of ink, it is desirable to consider as about 0.005 wt(s)% - 0.5wt% about 0.001 wt(s)% - 1wt%. These surfactants are contributed also on the wiper cleaning disposition of an ink-jet head.

[0026] Moreover, the ink for ink-jet record of this invention can contain bases, such as acids, such as a hydrochloric acid, a sulfuric acid, a nitric acid, an acetic acid, a citric acid, oxalic acid, a malonic acid, a boric acid, a phosphoric acid, phosphorous acid, and a lactic acid, a sodium hydroxide, a potassium hydroxide, a lithium hydroxide, and ammonia, as a pH regulator, and can contain phosphate, an oxalate, an amine salt, a good buffer, etc. as a buffer. pH of the ink for this ink-jet record -- the preservation stability of ink and a head, and a cartridge -- it is desirable pH 4-12 and to be especially referred to as pH 5-11 in consideration of the corrosion of a member
[0027] Moreover, as a solubilizing agent, polyethyleneimine, polyamine, a polyvinyl pyrrolidone, a polyethylene glycol, a cellulosic, etc. can be contained as physical-properties regulators, such as an acetamide and a betaine, and it can contain cyclodextrin, poly cyclodextrin, large annular amines, and crown ethers as a clathrate compound, and the ink for ink-jet record of this invention contains an antifungal agent, a rusr-proofer, a germicide, an antioxidant, a chelating agent, a DENDO reamer, a polymer emulsion, etc., and can carry out the thing of them if needed further.
[0028] It is characterized by the drying time when the ink for ink-jet record of this invention usually prints the solid picture of 100% of rates of picture area in the paper being less than 5 seconds. The solid picture of 100% of rates of picture area in this invention is a solid picture which has the amount of ink per unit area in the range of 2 - 2.0 mg/cm2 1.2mg [/cm ] abbreviation. surface tension [ in / 12 or less and 25 degrees C / as mentioned above / drying time / this / for less than 5 seconds / in an S.P. value ] -- the water-soluble organic compound of less than 40 mN/m -- 3.0wt(s)% - 15.0wt% -- it can attain by containing
[0029] Moreover, as for the steady flow viscosity of the ink for ink-jet record of this invention, it is desirable that it is the range of 1.8mPas - 4.0mPas. 1. Since it becomes easy to move a pigment particle at the same time when it comes to less than 8 mPases it falls from a nozzle and becomes easy to produce omission, it becomes easy to produce condensation. Moreover, if 4.0mPas(es) are exceeded, the resistance to the regurgitation force will become large.
[0030] moreover, the conductivity of the ink for ink-jet record of this invention -- 0.03S/m- it is desirable especially that it is the range of 0.05 - 0.3 S/m 0.4 S/m If when it comes to less than 0.03 S/m the maceration on the front face of a pigment in which self-distribution is possible is inadequate, dispersibility becomes bad and 0.4 S/m is exceeded, the electric double layer of the circumference of a pigment particle will become thin, the distance between particles will become short, and the dispersibility of a pigment will get worse. Therefore, it is desirable to press down electrolytes other than the pigment in which self-distribution is possible to necessary minimum

so that too highly [ ink conductivity ].
[0031] In addition, since the content of Mg and Fe in ink is in the inclination to which the burn on a heater is made to increase when it increased, and it promotes condensation of a pigment and also the record method of making ink breathing out by operation of heat energy is used, it is desirable to be referred to as less than 5 ppm. removal of Mg and Fe is independent in adsorption by filtration by rinsing, a reverse osmosis membrane/ultrafiltration membrane, etc., use of ion exchange resin, activated carbon/zeolite, etc. — or it can carry out by combining Moreover, what is necessary is just to remove Mg and Fe by the effective and suitable method in the state of each pigment itself / pigment dispersion liquid / ink, in order to mainly mix from a pigment.
[0032] Hereafter, the manufacture method of the ink for ink-jet record of this invention is explained. In manufacture of the ink for ink-jet record of this invention, in order to prevent mixing of the above-mentioned metal, it is desirable to include the process which distributes a pigment to underwater [ by the ultrasonic homogenizer or the high-pressure homogenizer ], without using distributed media. Furthermore, it is desirable to include the process which removes a big and rough particle by centrifugal separation. If the particle-diameter >0.5micrometer particle number contained in ink increases as described above, although the burn on distributed destabilization or a heater will be increased, a big and rough particle is efficiently removable by using the big and rough particle removal process by centrifugal separation. Moreover, in case centrifugal separation work is done, pigment concentration has the lower one effective for big and rough particle removal.
[0033] Therefore, as for the manufacture method of the ink for ink-jet record of this invention, it is desirable to include the process underwater distributed with an ultrasonic homogenizer or a high-pressure homogenizer, without using distributed media for the pigment in which self-distribution in water is possible, the distributed pigment, and the process which mixes the water which is the other materials, the above-mentioned water-soluble organic solvent, the above-mentioned water-soluble organic compound, etc., and it is desirable to include further the process which removes the big and rough particle by centrifugal separation.
[0034] Hereafter, the ink-jet record method of this invention is explained. The ink-jet record method of this invention is characterized by making the ink for ink-jet record of this invention breathe out from an orifice according to a record signal, and recording on a recorded material. Moreover, if heat energy is made to act on ink and ink is made to breathe out, since the effect of improving the burn on a heater will be acquired, it is desirable. Moreover, the effect which the amount of drops is stabilized and raises the regurgitation stability at the time of being the continuation regurgitation is acquired by forming the drop of one ink by two or more pulse impression, and making it breathe out. Furthermore, the effect of quality of image and a drying-property improvement comes to show up more greatly, so that ink-jet record resolution is made high and it is made small drops printing. 20 or less ngs are desirable still more desirable, and especially the discharge quantity per 1 drops is 15nm or less. Since the specific surface area of drops becomes large, this is presumed to be for a surface effect to show up strongly.
[0035] Moreover, high resolving, high concentration, and the picture of high fixing can be further acquired by mixing the ink for ink-jet record and the binder of this invention on a recorded material. A binder is the material for suppressing osmosis of the color material in ink, for example, polyvalent metallic salt, an organic amine and its salt, quarternary ammonium salt, cation nature polymer, Nonion nature polymer, anionic polymer, etc. can be used for it. What is necessary is for these binders to be gestalten, such as solution, and just to apply them to a recorded material simultaneously with ink record before and after ink record. As the method of an application, the method of making breathe out from an orifice according to a signal, and applying to a recorded material is effective and efficient like ink.
[0036] As mentioned above, in this invention, it sets in the ink for ink-jet record which contains the pigment in which self-distribution is possible in water, the water-soluble organic solvent, and water. (Operation) S. — surface tension [ in / 25 degrees C / value / P. / in 12 or less ] — the water-soluble organic compound of less than 40 mN/m — the weight of ink — being based — 3.0 - 15.0wt% — by containing The ink for ink-jet record whose drying time at the time of usually

printing the solid picture of 100% of rates of picture area in the paper is less than 5 seconds can be obtained. The ink for ink-jet record of this invention has a quick drying property in the paper, water resistance and scratch-proof nature are excellent, mothball stability is good, a quality of printed character is also good, and problems, such as blinding, do not arise.

[0037] The following is presumed although the mechanism of these effects acquired when surface tension [ in / 25 degrees C / in an S.P. value ] adds combining the water-soluble organic compound of less than 40 mN/m or less by 12, the pigment in which self-distribution in water is possible, and is not solved enough. That is, when usual pigment-content powder ink is used about the distributed stability of ink, it is made to distribute without making the pigment which is water-insoluble nature condense / sediment by the work of a dispersant which stuck to the pigment. On the other hand, when the pigment in which self-distribution is possible is used for the water of this invention, self-distribution is carried out in the state without a dispersant. Although possibility of having separated from the pigment to which the dispersant is sticking gradually, and having caused condensation/sedimentation of a pigment can be considered if the interaction of a penetrating agent and a dispersant is strong when penetrating agents exist mostly in ink, in the case of the pigment in which self-distribution is possible, there are such no worries in water. Moreover, since the particle diameter of a pigment is large and it cannot pass the opening in a recorded material easily compared with a color even if it uses the same penetrating agent about a drying property and a quality of printed character, too much osmosis is suppressed, and even if the drying time is early, it is thought that there are few blots. Moreover, surface tension becomes 40 or less mN/m, and scratch-proof nature is considered that wetting is good, the touch area of ink and a recorded agent spreads and fixing nature is improving by the bird clapper, although wetting to a recorded agent is bad in the case of the ink of high surface tension. Furthermore, since there is no bad influence by the interaction of a dispersant and a penetrating agent about blinding compared with usual pigment-content powder ink when the rate of a penetrating agent increases on a nozzle front face, it is thought that it becomes advantageous.

[0038]

[Example] Hereafter, an example explains this invention in more detail.

(Example 1) Microjet Black After diluting CW-1 (Orient company make) with water so that carbon concentration may become 10wt(s)%, centrifugal separation processing (7000r.p.m., 30 minutes) was carried out, and pigment dispersion liquid (carbon concentration 8.3wt%) were obtained.

The above-mentioned pigment dispersion liquid 50 weight sections glycerol (S. P. value : about 20) 15 weight sections diethylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=34 mN/m) 5 weight sections N, N-screw (2-hydroxyethyl)-2-aminomethyl ethane sulfonic acid 0.5 weight section NaOH 0.1 weight sections pure water Each component of the 29.4 weight sections above. It mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared.

[0039] (Example 2) Centrifugal separation processing (8000r.p.m., 40 minutes) of Cab-o-jet -300 (Cabot Corp. make) was carried out, and pigment dispersion liquid (carbon concentration 14.4wt%) were obtained.

The above-mentioned pigment dispersion liquid 35 weight sections ethylene glycol (S. P. value : about 18) 10 weight sections propylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=26 mN/m) 7 weight sections urea 5 weight sections pure water Each component of 43 weight sections above. It mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared.

[0040] (Example 1 of comparison) Not using the diethylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=34 mN/m), ink was prepared like the example 1 except having made the amount of pure water into the 34.4 weight sections. In addition, the S.P. value of a glycerol is 20 and is gamma=63 mN/m.

[0041] (Example 2 of comparison) Ink was prepared like the example 2 except having made the amount of the propylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=26 mN/m) into the 0.5 weight section, and having made the amount of pure water into 50 weight sections.

[0042]
(Example 3 of comparison)
Carbon black (Black Pearl L) 5 weight sections styrene-maleic-acid Na copolymer 1 weight section glycerol (S. P. value : about 20) 15 weight sections diethylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=34 mN/m) 5 weight sections N, N-screw (2-hydroxyethyl)-2-aminomethyl ethane sulfonic acid The 0.5 weight section NaOH 0.1 weight sections pure water A 73.4 weight sections carbon black and styrene-maleic-acid Na copolymer and purity. After preparing carbon black dispersion liquid, the component of above others was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared.
[0043]
(Example 4 of comparison)
Carbon black (Mitsubishi MA-100) A 4 weight sections styrene-maleic-acid Na copolymer 1 weight section glycerol (S. P. value : about 20, gamma=63 mN/m) 15 weight sections N, N-screw (2-hydroxyethyl)-2-aminomethyl ethane sulfonic acid The 0.5 weight section NaOH 0.1 weight sections pure water A 79.4 weight sections carbon black and styrene-maleic-acid Na copolymer and pure water. After preparing carbon black dispersion liquid, the component of above others was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared.
[0044] (Example 1 of an examination) Following the (1) - (11) was evaluated using the ink obtained in examples 1 and 2 and the examples 1-4 of comparison.
(1) In the ink surface tension of 23 degrees C, and the environment of 55%RH, it measured using the UIRU helmet I type surface tension balance.
(2) In the ink viscosity of 23 degrees C, and the environment of 55%RH, it measured by shear-rate 1,400S-1.
(3) In the ink conductivity of 23 degrees C, and the environment of 55%RH, it measured using the conductivity meter (product made from AOL-40:DKK).
[0045] (4) Set at the diameter of ink number-average part granule application, and 23 degrees C of distributed particle size distributions, and it is Microtrup. UPA:Leeds & It measured using the product made from Northrup.
(5) Set at 23 degrees C of >0.5micrometer particle numbers contained in ink, and it is Accusizer. TM770 Optical Particle Sizer:Particle Sizing The acquired value was converted into the value per ink 1dm3 using the product made from Systems.
[0046] (6) 1 / 4tone (2035x128 pulse) was made to breathe out 3 times on the frequency of 6kHz using a trial production head (400dpi) in the amount of ink drops of 23 degrees C, and the environment of 55%RH, weight was measured in response to ink to the wafer of an ink absorber, and the regurgitation weight of 1 drops was found by calculation.
(7) Time after printing and printing the solid picture of 100% of 10mmx50mm rates of picture area in the paper using the thermal ink jet printer of resolution 600dpi made as an experiment to evaluation in drying-time test 23degree C and the environment of 55%RH, using FX-L paper (Fuji Xerox make) as a typical regular paper until a drop disappears from in the paper visually was measured.
[0047] (8) typical using the thermal ink jet printer of resolution 400dpi made as an experiment to picture quality test evaluation — the printing test of 1dot line and a solid picture was usually performed in the paper, using FX-L paper (Fuji Xerox make) as paper As evaluation criteria, the homogeneity of a blot of a line and the edge of a solid picture was investigated, and evaluation was performed on the following criteria.
a) Line blot O [ ..... It is mustache-like b solid homogeneity with blot O in many portions. / ..... Disorder-less ** / ..... It is x with disorder slightly. / ..... Smoothness is missing by backlash backlash. ] ..... Blot-less ** ..... It spreads, is slightly and is x. [0048] (9) After printing the picture and passing after printing on the 1st using the thermal ink jet printer of resolution 400dpi made as an experiment to abrasion-resistance test evaluation, the picture section was ground against the finger and the following criteria estimated the dirt to the decoloring condition of the picture section, and the non-picture section.
O ..... [ ..... Picture concentration falls and the dirt of the non-picture section is conspicuous. ]

The fall of picture concentration is not seen but the non-picture section is also completely dirt-less **..... Most picture concentration is x which has dirt in the non-picture section a little although it is not falling. [0049] (10) When it was left in 23 degrees C and the environment of 55% RH and regurgitation resumption was carried out in the state where it does not act as a cap, after a regurgitation halt using the thermal ink jet printer of resolution 400dpi made as an experiment to blinding-proof nature test evaluation, neglect time until picture disorder arises was measured. Evaluation was performed on the following criteria.
O ..... [ ..... Less than 0.5 minutes. ] More than 1 minute ** ..... 0.5 minutes - 1 minute x [0050] (11) After saving for one month in ink preservation stability test 60degree C and -20 degrees C, re-filtration with 1-micrometer filter was performed, and the ink after re-filtration was printed using the thermal ink jet printer of resolution 400dpi. Evaluation was performed on the following criteria.
O ..... With [ it is after preservation and / filterability ] no change. Picture-concentration-change-less ** ..... Although the fall of filtration velocity is seen a little [ after / preservation ], picture concentration is change-less x..... Filter plugging of the ink after preservation was intense, and picture concentration fell greatly.
[0051]
[Table 1]
The table 1 ---------- An example The example of comparison ----------
1 2 1 2 3 4
Ink surface tension (mN/m) 37 34 6037 37 57 ---------- Ink viscosity (mPas) 3.0 2.8 2.32.3 3.4 2.5 ---------- Ink conductivity (S/m) 0.12 0.18 0.16 0.22 0.15 0.15 ---------- Diameter of ink number-average part granule application (nm) 27 41 2540 3748 ---------- A particle size distribution 1.8 2.2 1.8 2.02.7 2.3 ---------- >0.5-micrometer particle number (x1010) 0.4 2.4 0.5 2.0 12.52.5 ---------- The amount of ink drops (ng) 48 52 51 49 46 49 ---------- drying-time test (second) 2 1 40 15 2 40 ----------
Picture quality test a O O O x O O ---------- Picture quality test bO O O O O ---------- A wear-resistant test O Ox **O x ---------- A blinding-proof nature test O O O O xO ---------- Ink preservation stability test O O O O x O ----------
[0052] (Example 3) Microjet Black 5-micrometer filter after carrying out centrifugal separation processing (8000r.p.m., 20 minutes) of CW-1 (Orient company make) -- subsequently pressure filtration was carried out with 2-micrometer filter, and pigment dispersion liquid (carbon concentration 15.7wt%) were obtained
The above-mentioned carbon black dispersion liquid . 25 weight sections triethylene-glycol monobutyl ether (S. P. value : about 10, gamma=34 mN/m) 4 weight sections propylene glycol (S. P. value : about 16) 10 weight sections SAFI Norian 440 (HLB 6 [ about ]) 0.3 weight sections urea 3 weight sections pure water The 57.7 weight sections. [0053] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 2.6mPas, 29 mN/m, 0.09 S/m, 24nm, 1.9, and 1.1x1010 pieces, respectively, and the amounts of drops were 51ng(s). All the results of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test of the drying time were O for 3 seconds.
[0054] (Example 4) After processing Cab-o-jet -300 (Cabot Corp. make) with an ultrasonic homogenizer for 30 minutes, centrifugal separation processing (7000r.p.m., 20 minutes) was diluted and carried out to 10% concentration with water, and pigment dispersion liquid (8.8% of carbon concentration) were obtained.
The above-mentioned carbon black dispersion liquid . 50 weight sections diethylene glycol (S. P. value : about 15) 10 weight sections N-methyl-2-pyrrolidone (S. P. value : about 11, gamma=48 mN/m) 5 weight sections dipropylene-glycol monobutyl ether (S. P. value : about 10, gamma=29

mN/m) 5 weight sections pure water 30 weight sections. [0055] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 2.5mPas, 33 mN/m, 0.18 S/m, 25nm, 2.1, and 1.6x1010 pieces, respectively, and the amounts of drops were 49ng(s). All the results of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test of the drying time were O for 1.5 seconds.
[0056] (Example 5) After carrying out scaling processing of the carbon black (Raven 5750) by the hypochlorous acid Na, it adjusted after [ pH / 7.5 ] desalting and distributed by using pure water as a solvent using the ultrasonic homogenizer. After distribution, centrifugal separation processing (7000r.p.m., 20 minutes) was carried out, and pigment dispersion liquid (11% of carbon concentration) were obtained.
The above-mentioned pigment dispersion liquid . 40 weight sections triethylene glycol (S. P. value : about 14) 10 weight sections propylene-glycol monopropyl ether (S. P. value : about 11, gamma=26 mN/m) 8 weight sections polyoxyethylene alkyl ether 0.3 weight sections pure water The 41.7 weight sections. [0057] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 2.5mPas, 36 mN/m, 0.17 S/m, 59nm, 2.1, and 2.8x1010 pieces, respectively, and the amounts of drops were 47ng(s). For the result of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test, the drying time was O for 2 seconds.
[0058] (Example 6) After carrying out graft-polymer processing of styrene sulfonic-acid Na to carbon black (Mitsubishi MA-100), it distributed using the ultrasonic homogenizer by using pure water as a solvent, and after that, centrifugal separation processing (8000r.p.m., 40 minutes) was carried out, and pigment dispersion liquid (carbon concentration 7.8wt%) were obtained.
The above-mentioned pigment dispersion liquid . 60 weight sections thiodiethanol 20 weight sections polyoxyethylene polyoxypropylene blockpolymer 0.1 weight sections diethylene-glycol monochrome hexyl ether (S. P. value : about 10, gamma=29 mN/m) 3 weight sections pure water The 16.9 weight sections [0059] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 2.6mPas, 33 mN/m, 0.15 S/m, 47nm, 2.4, and 2.4x1010 pieces, respectively, and the amounts of drops were 53ng(s). All the results of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test of the drying time were O for 1 second.
[0060] (Example 7) After making it distribute by using water as a solvent using a high-pressure homogenizer so that carbon concentration may become 20% about the carbon black (Special Bk 4A) which performed plasma treatment, centrifugal separation processing (7000r.p.m., 30 minutes) was carried out, and pigment dispersion liquid (carbon concentration 15.6wt%) were obtained.
The above-mentioned pigment dispersion liquid . 5 weight sections trimethylol propane 15 weight sections glycerol (S. P. value : about 20) 5 weight sections propylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=26 mN/m) 5 weight sections polyoxyethylene perfluoro alkyl ether (HLB 13 [ about ]) 0.01 weight sections pure water 70 weight sections. [0061] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 3.3mPas, 23 mN/m, 0.14 S/m, 44nm, 2.3, and 1.7x1010 pieces, respectively, and the amounts of drops were 44ng(s). All the results of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test of the drying time were O for 2 seconds.
[0062]

(Example 8)
Pigment dispersion liquid used in the example 2 . 30 weight sections 1,5-pentanediol (S. P. value : about 12.5) 10 weight sections dipropylene-glycol monoethyl ether (S. P. value : about 10.5, gamma=28 mN/m) 8 weight sections isopropyl alcohol 3 weight sections silicone polyoxyethylene addition product (HLB 7 [ about ]) 0.1 weight sections polyoxyethylene oleyl ether (HLB 12 [ about ]) 0.3 weight sections pure water The 48.6 weight sections. [0063] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 3.0mPas, 33 mN/m, 0.16 S/m, 40nm, 2.4, and 1.3x1010 pieces, respectively, and the amounts of drops were 31ng(s). Moreover, for the result of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test, the drying time was O for 4 seconds. In addition, the amount of drops measured discharge quantity on the same conditions as an example 1 using the trial production head of 600dpi.
[0064]
(Example 9)
Pigment dispersion liquid used in the example 1 . 50 weight sections dipropylene glycol (S. P. value : about 13) 15 weight sections diethylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=34 mN/m) 5 weight sections benzoic acid Na 0.5 weight sections styrene-acrylic-ester-acrylic-acid emulsion (average molecular weight 12000, acid number 20 [ about ]) 1.0 weight sections pure water The 28.5 weight sections. [0065] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The amounts of drops were 31ng(s) in the viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution and a >0.5micrometer particle number, 3.2mPas, 36 mN/m, 3.2mPas, 0.22 S/m, 40nm, 2.4, and 1.5x1010 pieces. Moreover, for the result of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test, the drying time was O for 1.5 seconds. In addition, the amount of drops measured discharge quantity on the same conditions as an example 1 using the trial production head of 600dpi.
[0066]
(Example 10)
Pigment dispersion liquid used in the example 3 . 30 weight sections glucose 10 weight sections 1,5-pentanediol (S. P. value : about 12.5) 10 weight sections dipropylene-glycol monopropyl ether (S. P. value : about 10.5, gamma=28 mN/m) 12 weight sections SAFI Norian 104 0.1 weight sections N-(2-acetamide) iminodiacetic acid The 0.5 weight sections LiOH 0.1 weight sections pure water The 37.3 weight sections. [0067] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 3.4mPas, 30 mN/m, 0.22 S/m, 25nm, 2.1, and 3.9x1010 pieces, and the amounts of drops were 45ng(s). For the result of 1s, a picture quality test, an abrasion-resistance test, and a blinding-proof nature test, the drying time was O, and the result of an ink preservation stability test was **.
[0068] (Example 11) After making it distribute by using water as a solvent using a high-pressure homogenizer so that it may become 20% of pigment concentration about a plasma treatment phthalocyanine pigment, centrifugal separation processing (7000r.p.m., 30 minutes) was carried out, and activated-charcoal-absorption processing was performed, and it filtered with 5-micrometer filter after that, and pigment dispersion liquid (14.7% of phthalocyanine-pigment concentration) were obtained.
The above-mentioned pigment dispersion liquid . 25 weight sections glycerol (S. P. value : about 20) 15 weight sections dipropylene-glycol monobutyl ether (S. P. value : about 10, gamma=29 mN/m) 5 weight sections polyoxyethylene polyoxypropylene block copolymer (HLB 10 [ about ]) The 0.3 weight sections PROXEL GXL0.03 weight section pure water The 53.7 weight sections.
[0069] Each above-mentioned component was mixed enough, pressure filtration was carried out

with 2-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 2.8mPas, 32 mN/m, 0.22 S/m, 87nm, 2.4, and 5.7x1010 pieces, and the amounts of drops were 46ng(s). For the result of 1.5 seconds, a picture quality test, an abrasion-resistance test, and a blinding-proof nature test, the drying time was O, and the result of an ink preservation stability test was **.

[0070] (Example 12) Ink was prepared by the same composition and the same method as an example 11 except not performing centrifugal separation processing and activated-charcoal-absorption down stream processing of an example 11. The viscosity of ink was [ 32 mM/m, conductivity, the diameter of number-average part granule application particle size distribution, and the >0.5micrometer particle number of 2.8mPas(es) and surface tension ] 0.24 S/m, 110nm, 3.2, and 9.0x1010 pieces, respectively, and 15 ppm, 10 ppm, and the amounts of drops of the content of Fe and Mg were 46ng(s), respectively. The drying time was 1.5 seconds and, for the result of O, an abrasion-resistance test, an ink preservation stability test, and a blinding-proof nature test, the result of a picture quality test was **.

[0071]

(Example 5 of comparison)

Pigment dispersion liquid used in the example 1 . 50 weight sections diethylene glycol 15 weight sections tetraethylene-glycol monomethyl ether (S. P. value : about 10.5, gamma=50 mN/m) 5 weight sections thiourea 5 weight sections pure water 25 weight sections. [0072] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink was [ 47 mN/m, conductivity, the diameter of number-average part granule application particle size distribution, and the >0.5micrometer particle number of 2.7mPas(es) and surface tension ] 0.14 S/m, 28nm, 1.9, and 0.8x1010 pieces, respectively, and the amounts of drops were 53ng(s). Although the result of a picture quality test, a blinding-proof nature test, and an ink preservation stability test was O, for the result of an abrasion-resistance test, the drying time was x for 30 seconds.

[0073]

(Example 6 of comparison)

Pigment dispersion liquid used in the example 2 35 weight sections diethylene glycol 5 weight sections propylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=26 mN/m) 20 weight sections pure water Each component of 40 weight sections above. It mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 3.2mPas, 33 mN/m, 0.16 S/m, 44nm, 2.8, and 7.5x1010 pieces, respectively, and the amounts of drops were 48ng(s). Although the result of the drying time of an abrasion-resistance test and a blinding-proof nature test was O for 1 second, the result of a picture quality test and an ink preservation stability test was x.

[0074]

(Example 13)

Pigment dispersion liquid used in the example 4 50 weight sections 1 and 2, 6-hexane triol (S. P. value : about 15) 10 weight sections ethylene glycol monopropyl ether (S. P. value : about 11, gamma=32 mN/m) 10 weight sections oxyethylene oleyl ether (HLB 10 [ about ]) 0.2 weight sections pure water The 29.8 weight sections. [0075] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 2.5mPas, 39 mN/m, 0.13 S/m, 20nm, 2.1, and 1.2x1010 pieces, and the amounts of drops were 27ng(s). In addition, the amount of drops measured discharge quantity, in addition the amount of drops on the same conditions as an example 1 using the trial production head of 600dpi. A picture quality test, the abrasion-resistance test, the blinding-proof nature test, the drying-time test, and the ink preservation stability test were carried out using the thermal ink jet printer of resolution 600dpi which adopted the method of forming one drop by impressing the driving signal which consists of idle periods between the main pulse made as an experiment to evaluation, a pre pulse

and a pre pulse, and a main pulse. All the other test results of the drying time were O in 4 seconds.
[0076] (Example 14) The ink of the following composition was prepared.
– Cyanogen ink Projet Fast Product made from Cyan.2:ZENECA 4 weight sections butyl carbitol 5 weight sections thiodiethanol 15 weight sections pure water Each component of 76 weight sections above. The mixed dissolution was carried out enough, pressure filtration was carried out with 0.45–micrometer filter, and ink was prepared.
[0077] – Magenta ink Projet Fast Instead of made from Cyan.2:ZENECA, it is Projet. Fast Magenta Except having used 2, the mixed dissolution of each component was enough carried out by the same composition, pressure filtration was carried out with 0.45–micrometer filter, and ink was prepared.
– Yellow ink Projet Fast Instead of made from Cyan.2:ZENECA, it is Projet. Fast Yellow Except having used 2, the mixed dissolution of each component was enough carried out by the same composition, pressure filtration was carried out with 0.45–micrometer filter, and ink was prepared.
[0078] (Example 2 of an examination)
(12) The following evaluation was carried out using the ink of the heavy picture quality test example 1 and the cyano ink obtained in the example 14, Magenta ink, and yellow ink.
[0079] The printing test of the solid picture pattern with which a black 1dot line [ as opposed to / usually using FX–L paper (Fuji Xerox make) as paper / a color background to in the paper / the ] and typical each color adjoin was performed using the thermal ink jet printer of resolution 400dpi made as an experiment to evaluation. As evaluation criteria, a blot of a line and the homogeneity of the solid picture contiguity section were investigated, and evaluation was performed on the following criteria.
[0080] a) Line blot O [ ..... It is mustache–like b solid homogeneity with blot O in many portions. / ..... Disorder–less ** / ..... It is x with disorder slightly. / ..... If smoothness was missing at all by backlash backlash, the evaluation result of a and b was O also about which ink. ] ..... Blot–less ** ..... It spreads, is slightly and is x.
[0081] (Example 3 of an examination) The following evaluation was carried out using the ink of an example 1, and the binder of the following composition.
– A binder A styrene–maleic–acid Li copolymer 5 weight sections ethylene glycol 15 weight sections polyoxyethylene oleyl ether 0.2 weight section isopropyl alcohol 3 weight sections pure water The thermal ink jet printer of resolution 400dpi made as an experiment to 76.8 weight sections evaluations is used, and it is a typical regular paper. Using FX–L paper (Fuji Xerox make), Binder A was piled up in the paper, the ink of an example 1 was piled up within 5 seconds after the regurgitation, it was made to print and the picture quality test of the above (9) was carried out. The result of a picture quality test is O and showed picture concentration higher than an example 1.
[0082] (Example 4 of an examination) The following evaluation was carried out using the ink of an example 2, and the binder of the following composition.
– Binder B benzalkonium chloride 1 weight section poly allylamine hydrochloride 5 weight sections glycerol 15 weight sections pure water The same picture quality test as the example 3 of 79 weight sections examinations was carried out. The result of a picture quality test is O and showed picture concentration higher than an example 2.
[0083] (Example 5 of an examination) The following evaluation was carried out using the ink of an example 1. The discharge quantity per 1 drops and the drying time were found like (6) of the example 1 of an examination, and (7) except having used the ink jet printer of 800dpi made as an experiment to evaluation. Moreover, the character of eight points was printed on FX–L paper (Fuji Xerox make) as a typical regular paper using the same printer. It excelled compared with the time of printing by the printer which 1 second and character grace used the discharge quantity per 1 drops by 13ng(s), and used the drying time by (8) of the example 1 of an examination.
[0084]
[Effect of the Invention] The ink for ink–jet record of this invention usually has quick dryness in

the paper, a quality of printed character, water resistance / scratch-proof nature, and mothball stability are excellent, and, moreover, blinding etc. does not happen.

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[Translation done.]

## TECHNICAL FIELD

[The technical field to which invention belongs] this invention relates to the ink for ink-jet record used for the recording devices (a printer, a copying machine, facsimile, a word processor, plotter, etc.) of an ink-jet method, its manufacture method, and the ink-jet record method using it.

[Translation done.]

## PRIOR ART

[Description of the Prior Art] A liquid or melting dry ink is breathed out from a nozzle, a slit, a porosity film, etc., the so-called recording device of the ink-jet method which records on recorded materials, such as paper, cloth, and a film, is small, and since it has advantages, such as cheapness and calm nature, various examination is performed. In recent years, not only the black monochrome printer by which the so-called good qualities of printed character, such as a report form and a copy paper, are obtained but many products which can perform full color record are usually marketed in the paper, and it came to occupy a big position in the field of the recording device.

[0003] The ink used by the ink-jet recording device usually mainly consists of a solvent, color material, and an additive. About the ink for ink-jet record, the blot in (1) paper, high resolution without a fogging, and a picture uniform at high concentration are acquired, (2) The blinding at the nose of cam of a nozzle by ink dryness does not occur, but there are demand properties, like that **** responsibility and **** stability are always good, that the robustness of that the drying property of the ink in the paper is good and (3) (4) picture is good, and (5) mothball stability is good.

[0004] Various meanses are provided as a remedy for fulfilling these demand properties. For example, the drying property of the ink in the paper of the above (3) is important in order to raise the speed of a recording device or to prevent the color mixture blot at the time of performing color printing, the high solvent of permeability or volatility is used, or the improvement by adding a surfactant etc. has been made conventionally.

[0005] Moreover, about the robustness of the picture of the above (4), many ink which has improved water resistance is indicated by using a pigment instead of a color as a color material (JP,56-147869,A, JP,2-255875,A, U.S. Pat. No. 5085698 specification, etc.). However, although water resistance improves when pigment ink is used, there is a fault that scratch-proof nature is inferior compared with color ink. Furthermore, when pigment ink is used, there is also a problem of becoming what is generally inferior compared with color ink about the mothball stability of the above (5).

[0006] The method of oxidizing carbon black is indicated by the method to which a carbon black front face is made to carry out the polymerization of the water-soluble monomer etc., and JP,8-3498,A at the method of carrying out azo coupling of the substituent which contains a water solubilization machine in carbon black to for example, a U.S. Pat. No. 5571311 specification as a method of solving the problem at the time of using a pigment, and JP,8-81646,A. However, mothball stability is good, a drying property in the paper is usually quick, a good quality of printed character is obtained, and the ink for ink-jet record which was moreover excellent in water resistance / scratch-proof nature is not yet found out.

## EFFECT OF THE INVENTION

[Effect of the Invention] The ink for ink-jet record of this invention usually has quick dryness in the paper, a quality of printed character, water resistance / scratch-proof nature, and mothball stability are excellent, and, moreover, blinding etc. does not happen.

[Translation done.]

## TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] Therefore, the technical problem of such a Prior art is solved, mothball stability is good, a drying property in the paper is usually quick, a good quality of printed character is obtained, and the purpose of this invention aims at offering the ink which was moreover excellent in water resistance / scratch-proof nature.

[Translation done.]

## MEANS

[Means for Solving the Problem] the water-soluble organic compound whose surface tension [ in / 25 degrees C / on the ink for ink-jet record containing the pigment which this invention persons can self-distribute in water, the water-soluble organic solvent, and water, and / in an S.P. value ] is less than 40 mN/m or less in 12 — the total weight of ink — being based — 3.0 - 15.0wt% — by containing, it found out that the above-mentioned purpose could be attained and this invention was completed

[0009] Namely, this invention is set in the ink for ink-jet record which contains the pigment and water-soluble organic solvent in which self-distribution is possible in water and water. S. Surface tension [ in / 25 degrees C / in P. value ] or less by 12 the water-soluble organic compound of less than 40 mN/m the total weight of ink — being based — 3.0 - 15.0wt% — the pigment in which self-distribution in the ink for ink-jet record and water which are characterized by containing is possible with the process underwater distributed using an ultrasonic homogenizer or a high-pressure homogenizer The manufacture method of the ink for ink-jet record characterized by the dispersed pigment, water, the water-soluble organic solvent, and an S.P. value including the process with which the surface tension in 25 degrees C mixes the water-soluble organic compound of less than 40 mN/m or less by 12, And the aforementioned ink is made to breathe out from an orifice according to a record signal, and it consists of the ink-jet record method characterized by recording on a recorded material.

[0010] Hereafter, this invention is explained in detail. Even if "the pigment in which self-distribution in water is possible" used in this invention does not have existence of a dispersant in the front face including many solubilization machines to water, it is a pigment distributed stably. In order to be "the pigment in which self-distribution in water is possible" of this invention Distributed equipments, such as an ultrasonic homogenizer, a nano mizer, a Micro fluidizer, and a ball mill, are used. When the pigment concentration of a supernatant is measured after having made water distribute a pigment, without using a dispersant so that it may become concentration (water 95wt%/pigment 5wt%), measuring initial pigment concentration and leaving dispersion liquid with a glass bottle after that on the 1st, the pigment concentration should just be 98% or more of initial concentration.

[0011] "The pigment in which self-distribution in water is possible" of this invention can be manufactured by performing surface-treatment processing of acid-base processing, coupling-agent processing, polymer graft processing, plasma treatment, or oxidization/reduction processing to the usual pigment. By performing such surface treatment, more solubilization machines to water than the usual pigment come to be included, and self-distribution is attained. As a usual pigment to which surface-treatment processing is performed Raven 5250 Raven 3500 Raven 5750 Raven 1080 Regal 330R, Mogul L, Monarch 1000 Color Black FW2, Black Pearl L, Printex V, Special Bk Although the carbon black of 4A and Mitsubishi No.25 grade can be mentioned as a desirable example C. I. Pigment Blue1 and C.I. Pigment Blue 3 and C.I. Pigment Blue 15 C.I. Pigment Blue 15-3, C.I. Pigment Blue 16 C.I. Pigment Blue The cyano pigment of the 60th grade, C. I. Pigment Red 5 and C.I. Pigment Red12 and C.I. Pigment Red 48 and C.I. Pigment Red 112 and C.I. Pigment Red 122 C.I. Pigment Red 146 C.I. Pigment Red 168 C.I. Pigment Red The Magenta pigment of the 202nd grade, C. I. Pigment Yellow 1 C.I. Pigment Yellow 2,

C.I.Pigment Yellow 3 C.I. Pigment Yellow 13 C.I. Pigment Yellow 16 C.I. Pigment Yellow 73 C.I. Pigment Yellow 83 C.I. Pigment Yellow 98 C.I. Pigment Yellow 114 C.I. Pigment Yellow 128 C.I. PigmentYellow The color pigment of the 154th grade It can use.

[0012] Moreover, "the pigment in which self-distribution in water is possible" of this invention can also use for commercial water the pigment in which self-distribution is possible as it is. As an example of the pigment of such marketing, they are Cab-o-jet -200, Cab-o-jet -300, IJX-55 (above, Cabot Corp. make), and Microjet. Black CW-1 (Orient chemistry company make) etc. is mentioned.

[0013] Although the solubilization machines to the water contained in the pigment in which self-distribution in water is possible may be Nonion nature, cation nature, and anionic any, a sulfonic acid, a carboxylic acid, a hydroxyl group, its phosphoric acid, etc. are mainly desirable. Although it can use also in the state of a free acid as it is in the case of a sulfonic acid, a carboxylic acid, and a phosphoric acid, it does not matter even if it forms the salt. When the salt is formed, as for the counter ion of an acid, generally, it is desirable that they are Li+, Na+, K+, NH4+, and an organic amine.

[0014] this invention -- setting -- the content of the pigment in which self-distribution in water is possible -- a total ink weight -- being based -- desirable -- 0.1 - 20wt% -- more -- desirable -- 0.5 - 15wt% It is 1 - 10wt% especially preferably. If the content of a pigment exceeds 20wt (s)%, it will become easy to produce the blinding in the nose of cam of a nozzle. Sufficient concentration will not be obtained if a content becomes less than [ 0.1wt% ]. [0015] Moreover, as for the diameter of number-average part granule application in the ink of the pigment in which self-distribution in water is possible, it is especially desirable the range of 15nm - 100nm and that it is the range of 15nm - 50nm. If the diameter of number-average part granule application is in this range, it will be hard to produce blinding and will become the thing excellent also in preservation stability. If the diameter of number-average part granule application is set to less than 15nm, probably because the surface area per particle unit volume will become large and the touch area between particles will become easy to become large in ink, ink viscosity becomes high and tends to produce blinding. On the contrary, if a mean particle diameter exceeds 100nm, distribution will tend to become unstable and the inclination which leads to condensation and sedimentation of a pigment will be seen.

[0016] Moreover, as for especially the distributed particle size distribution (ratio to the diameter of number-average part granule application of volume mean-dispersion particle size) of a pigment in which self-distribution in water is possible, it is [ 3.5 or less ] desirable that it is 2.5 or less. When distributed particle size distribution exceed 3.5, even if the diameter of number-average part granule application is in the above-mentioned range, there is an inclination which becomes easy to cause condensation and sedimentation by using some big particulate materials as a nucleus. "used in this invention -- S. -- P. value -- 12 or less -- surface tension -- water-soluble organic compound" of less than 40 mN/m -- the total weight of ink -- being based -- 3.0wt% - 15.0wt% -- the value of distributed particle size distribution can be made or less into 3.5 by containing

[0017] Furthermore, when using the ink-jet recording method characterized by making ink breathe out by operation of heat energy, in order to suppress the burn on a heater, it is desirable <=6x1010 piece / dm3, and to set especially to <=3x1010 piece / dm3 the particle-diameter >0.5micrometer particle number contained in ink. Particle-diameter > In order to make a 0.5-micrometer particle number into the above-mentioned range, in manufacture of ink, it is desirable to take in the big and rough particle removal process by centrifugal separation or filtration. If the particle-diameter >0.5micrometer particle number contained in ink exceeds 6x1010 pieces / dm3, it may become the cause of blinding or sedimentation of a pigment may arise during preservation, or the burn on a heater is accelerated and the fall of the amount of drops may arise. If a lot of surfactants are added in order to bring the drying time forward, although a particle number will tend to increase by the usual pigment-content powder system, it becomes possible to suppress a >0.5micrometer particle number by using the pigment in which self-distribution is possible.

[0018] An S.P. value (Solubility Parameter) is 12 or less thing, and, as for the water-soluble

organic compound used in this invention, it is desirable especially that they are or less 12 eight or more things. S.P. The drying time will become long if a value exceeds 12. Moreover, if an S.P. value becomes less than eight, it will become easy for the solubility to water to fall. Moreover, the surface tension in 25 degrees C is the thing of less than 40 mN/m, and, as for the water-soluble organic compound used in this invention, it is desirable especially that it is 20 or more mN/m the thing of less than 40 mN/m. If surface tension becomes 40 or more mN/m, the wettability to a recorded material will become bad and neither a drying property nor abrasion resistance will be improved. Moreover, since it gets wet too much to record material when it comes to less than 20 mN/m, it becomes easy to produce a blot and a strike-through. Moreover, less than 400 thing has [ the water-soluble organic compound used in this invention ] desirable molecular weight. If molecular weight becomes 400 or more, since the blinding nature of a nozzle will become bad, it is not desirable.

[0019] The compound which has the structure shown by R-O-XnH (the functional group chosen from the group which consists of the alkyl, the alkenyl, the alkynyl, the phenyl, alkylphenyl, and cycloalkyl of R:C1-C8, X:oxyethylene or oxypropylene, integer of n=1-8) from the point of pigment-content powder stability as a water-soluble organic compound which satisfies these requirements is desirable. Moreover, it is desirable in the above-mentioned formula that R is C3-C6 and n is especially the integer of 1-6. As an example of the compound which has the structure shown by the above-mentioned formula An ethylene glycol monobutyl ether, the ethylene glycol monopropyl ether, The diethylene-glycol monobutyl ether, the diethylene-glycol monochrome hexyl ether, The diethylene-glycol monopropyl ether, the dipropylene-glycol monopropyl ether, The dipropylene-glycol monobutyl ether, the propylene-glycol monobutyl ether, the propylene-glycol monopropyl ether, the triethylene-glycol monobutyl ether, a diethylene-glycol monochrome phenyl ether, etc. are mentioned. Also in these, the diethylene-glycol monobutyl ether, the diethylene-glycol monochrome hexyl ether, the propylene-glycol monobutyl ether, and the triethylene-glycol monobutyl ether are desirable.

[0020] such [ the ink for ink-jet record of this invention ] a water-soluble organic compound -- the total weight of ink -- being based -- 3.0wt% - 15.0wt% -- desirable -- 3.0wt(s)% - 10.0wt% -- it contains If a content becomes less than [ 3.0wt% ], the pass-through effect will seldom be discovered and a rate of drying will fall. On the contrary, if 15.0wt% is exceeded, the obstacle that become or the distributed stability of the pigment in which self-distribution is possible falls to water by too much osmosis that it is easy to produce a blot will arise.

[0021] The water-soluble organic solvent used in this invention Although it will not be limited especially if it is used in order to prevent evaporation of water in ink-jet ink For example, ethylene glycol, a diethylene glycol, a propylene glycol, A polyethylene glycol, a triethylene glycol, a glycerol, a trimethylol propane, Polyhydric alcohol, such as 1, 2, 6-hexane triol, 1,5-pentanediol, and a dipropylene glycol Saccharides, such as a glucose, a fructose, a galactose, a mannose, and a xylose, Thiodiethanol, 2-mercaptoethanol, a thio glycerol, Nitrogen-containing solvents, such as sulfur-containing solvents, such as a sulfolane and dimethyl sulfoxide, 2-pyrrolidone, a N-methyl-2-pyrrolidone, a cyclohexyl pyrrolidone, a monoethanolamine, a triethanolamine, and a diethanolamine, can be used. Also in these, ethylene glycol, a diethylene glycol, a propylene glycol, a glycerol, thiodiethanol, a sulfolane, 2-pyrrolidone, a N-methyl-2-pyrrolidone, etc. are desirable. Moreover, even if it uses these independently, they may mix and use two or more sorts. As for especially the content of the water-soluble organic solvent, based on the total weight of ink, it is desirable to consider as about 3 wt(s)% - 50wt% about 1 wt% - 60wt%. If 60wt (s)% is exceeded, ink viscosity will rise and **** stability and **** responsibility will fall. Moreover, when it comes to less than [ 1wt% ], suppressing [ of evaporation of water ] is easy to become inadequate, and it becomes easy to start blinding.

[0022] although especially the water used in this invention is not limited -- pure water -- desirable -- the total weight of ink -- being based -- 35wt% -- being contained -95wt is desirable If when it comes to less than [ 35wt% ] the distributed stability of the pigment in which self-distribution is possible gets worse in water, or ink viscosity may become high, **** stability may fall and 95wt% is exceeded, evaporation tends to increase at the nozzle edge and may produce blinding.

[0023] As a **** stabilizing agent, the ink for ink-jet record of this invention can mix independent or two sorts or more, and can contain a urea, thiourea, an ethylene urea, an ethylenetiourea, methylurea, a dimethyl urea, a methylthio urea, dimethyl thiourea, etc. As for especially the content of these **** stabilizing agents, it is desirable to the weight of ink to consider as about 1 wt% - 10wt% about 0.5 wt(s)% - 15wt%.
[0024] Moreover, the ink for ink-jet record of this invention As a surfactant, a Nonion nature activator, for example, polyoxyethylene alkyl phenyl ether, Polyoxyethylene alkyl ether, polyoxyethylene fatty acid ester, A sorbitan fatty acid ester, polyoxyethylene sorbitan fatty acid ester, Polyoxyethylene glycerine fatty acid ester, polyglyceryl fatty acid ester, Polyoxyethylene sorbitol fatty acid ester, a polyoxyethylene sterol, The polyoxyethylene polyoxypropylene ether, polyoxyethylene fatty acid amide, A polyoxyethylene polyoxypropylene block copolymer, a tetramethyl crepe-de-Chine diol, Anionic surface active agents, such as a tetramethyl crepe-de-Chine diol ethyleneoxide addition product, For example, alkylbenzene sulfonates, an alkylphenyl sulfonate, Alkylnaphthalenesulfonate, a higher-fatty-acid salt, the sulfate salt of higher-fatty-acid ester, The sulfonate of higher-fatty-acid ester, the sulfate salt of the higher-alcohol ether, and a sulfonate, The formalin condensate of a high-class alkyl sulfo succinic-acid salt and a naphthalene sulfonate, A polystyrene sulfonate salt, a polyacrylate, polyoxyethylene-alkyl-ether phosphate, The others which are an alkyl ether carboxylate, an alkyl sulfate, an acrylic-acid-acrylic-ester copolymer, etc., Silicone system surfactants and perfluoroalkyl carboxylates, such as a polysiloxane polyoxyethylene addition product, Biosurfactants, such as fluorine system surfactants, such as a perfluoroalkyl sulfonate and oxyethylene perfluoro alkyl ether, a SUPIKURISU pole acid, and a rhamnolipid, a lysolecithin, etc. can be contained. Independent or two sorts or more can be mixed and used for these surfactants.
[0025] As for HLB of a surfactant, it is desirable that it is the range of 5-25 in consideration of pigment-content powder stability. As for especially the addition of a surfactant, based on the total weight of ink, it is desirable to consider as about 0.005 wt(s)% - 0.5wt% about 0.001 wt(s)% - 1wt%. These surfactants are contributed also on the wiper cleaning disposition of an ink-jet head.

[0026] Moreover, the ink for ink-jet record of this invention can contain bases, such as acids, such as a hydrochloric acid, a sulfuric acid, a nitric acid, an acetic acid, a citric acid, oxalic acid, a malonic acid, a boric acid, a phosphoric acid, phosphorous acid, and a lactic acid, a sodium hydroxide, a potassium hydroxide, a lithium hydroxide, and ammonia, as a pH regulator, and can contain phosphate, an oxalate, an amine salt, a good buffer, etc. as a buffer. pH of the ink for this ink-jet record -- the preservation stability of ink and a head, and a cartridge -- it is desirable pH 4-12 and to be especially referred to as pH 5-11 in consideration of the corrosion of a member
[0027] Moreover, as a solubilizing agent, polyethyleneimine, polyamine, a polyvinyl pyrrolidone, a polyethylene glycol, a cellulosic, etc. can be contained as physical-properties regulators, such as an acetamide and a betaine, and it can contain cyclodextrin, poly cyclodextrin, large annular amines, and crown ethers as a clathrate compound, and the ink for ink-jet record of this invention contains an antifungal agent, a rusr-proofer, a germicide, an antioxidant, a chelating agent, a DENDO reamer, a polymer emulsion, etc., and can carry out the thing of them if needed further.
[0028] It is characterized by the drying time when the ink for ink-jet record of this invention usually prints the solid picture of 100% of rates of picture area in the paper being less than 5 seconds. The solid picture of 100% of rates of picture area in this invention is a solid picture which has the amount of ink per unit area in the range of 2 - 2.0 mg/cm2 1.2mg [/cm ] abbreviation. surface tension [ in / 12 or less and 25 degrees C / as mentioned above / drying time / this / for less than 5 seconds / in an S.P. value ] -- the water-soluble organic compound of less than 40 mN/m -- 3.0wt(s)% - 15.0wt% -- it can attain by containing
[0029] Moreover, as for the steady flow viscosity of the ink for ink-jet record of this invention, it is desirable that it is the range of 1.8mPas - 4.0mPas. 1. Since it becomes easy to move a pigment particle at the same time when it comes to less than 8 mPases it falls from a nozzle and becomes easy to produce omission, it becomes easy to produce condensation. Moreover, if 4.0mPas(es) are exceeded, the resistance to the regurgitation force will become large.

[0030] moreover, the conductivity of the ink for ink-jet record of this invention -- 0.03S/m- it is desirable especially that it is the range of 0.05 - 0.3 S/m 0.4 S/m If when it comes to less than 0.03 S/m the maceration on the front face of a pigment in which self-distribution is possible is inadequate, dispersibility becomes bad and 0.4 S/m is exceeded, the electric double layer of the circumference of a pigment particle will become thin, the distance between particles will become short, and the dispersibility of a pigment will get worse. Therefore, it is desirable to press down electrolytes other than the pigment in which self-distribution is possible to necessary minimum so that too highly [ ink conductivity ].
[0031] In addition, since the content of Mg and Fe in ink is in the inclination to which the burn on a heater is made to increase when it increased, and it promotes condensation of a pigment and also the record method of making ink breathing out by operation of heat energy is used, it is desirable to be referred to as less than 5 ppm. removal of Mg and Fe is independent in adsorption by filtration by rinsing, a reverse osmosis membrane/ultrafiltration membrane, etc., use of ion exchange resin, activated carbon/zeolite, etc. -- or it can carry out by combining Moreover, what is necessary is just to remove Mg and Fe by the effective and suitable method in the state of each pigment itself / pigment dispersion liquid / ink, in order to mainly mix from a pigment.
[0032] Hereafter, the manufacture method of the ink for ink-jet record of this invention is explained. In manufacture of the ink for ink-jet record of this invention, in order to prevent mixing of the above-mentioned metal, it is desirable to include the process which distributes a pigment to underwater [ by the ultrasonic homogenizer or the high-pressure homogenizer ], without using distributed media. Furthermore, it is desirable to include the process which removes a big and rough particle by centrifugal separation. If the particle-diameter >0.5micrometer particle number contained in ink increases as described above, although the burn on distributed destabilization or a heater will be increased, a big and rough particle is efficiently removable by using the big and rough particle removal process by centrifugal separation. Moreover, in case centrifugal separation work is done, pigment concentration has a method of a low effective for big and rough particle removal.
[0033] Therefore, as for the manufacture method of the ink for ink-jet record of this invention, it is desirable to include the process underwater distributed with an ultrasonic homogenizer or a high-pressure homogenizer, without using distributed media for the pigment in which self-distribution in water is possible, the distributed pigment, and the process which mixes the water which is the other materials, the above-mentioned water-soluble organic solvent, the above-mentioned water-soluble organic compound, etc., and it is desirable to include further the process which removes the big and rough particle by centrifugal separation.
[0034] Hereafter, the ink-jet record method of this invention is explained. The ink-jet record method of this invention is characterized by making the ink for ink-jet record of this invention breathe out from an orifice according to a record signal, and recording on a recorded material. Moreover, if heat energy is made to act on ink and ink is made to breathe out, since the effect of improving the burn on a heater will be acquired, it is desirable. Moreover, the effect which the amount of drops is stabilized and raises the regurgitation stability at the time of being the continuation regurgitation is acquired by forming the drop of one ink by two or more pulse impression, and making it breathe out. Furthermore, the effect of quality of image and a drying-property improvement comes to show up more greatly, so that ink-jet record resolution is made high and it is made small drops printing. 20 or less ngs are desirable still more desirable, and especially the discharge quantity per 1 drops is 15nm or less. Since the specific surface area of drops becomes large, this is presumed to be for a surface effect to show up strongly.
[0035] Moreover, high resolving, high concentration, and the picture of high fixing can be further acquired by mixing the ink for ink-jet record and the binder of this invention on a recorded material. A binder is the material for suppressing osmosis of the color material in ink, for example, polyvalent metallic salt, an organic amine and its salt, quarternary ammonium salt, cation nature polymer, Nonion nature polymer, anionic polymer, etc. can be used for it. What is necessary is for these binders to be gestalten, such as solution, and just to apply them to a recorded material simultaneously with ink record before and after ink record. As the method of

an application, the method of making breathe out from an orifice according to a signal, and applying to a recorded material is effective and efficient like ink.

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[Translation done.]

## OPERATION

(Operation) as mentioned above, surface tension [ in / 25 degrees C / value / S.P. / on the ink for ink-jet record which contains the pigment in which self-distribution is possible in water, the water-soluble organic solvent, and water in this invention, and / in 12 or less ] -- the water-soluble organic compound of less than 40 mN/m -- the weight of ink -- being based -- 3.0 - 15.0wt% -- containing The ink for ink-jet record whose drying time at the time of usually printing the solid picture of 100% of rates of picture area in the paper is less than 5 seconds can be obtained. The ink for ink-jet record of this invention has a quick drying property in the paper, water resistance and scratch-proof nature are excellent, mothball stability is good, a quality of printed character is also good, and problems, such as blinding, do not arise.
[0037] The following is presumed although the mechanism of these effects acquired when surface tension [ in / 25 degrees C / in an S.P. value ] adds combining the water-soluble organic compound of less than 40 mN/m or less by 12, the pigment in which self-distribution in water is possible, and is not solved enough. That is, when usual pigment-content powder ink is used about the distributed stability of ink, it is made to distribute without making the pigment which is water-insoluble nature condense / sediment by the work of a dispersant which stuck to the pigment. On the other hand, when the pigment in which self-distribution is possible is used for the water of this invention, self-distribution is carried out in the state without a dispersant. Although possibility of having separated from the pigment to which the dispersant is sticking gradually, and having caused condensation/sedimentation of a pigment can be considered if the interaction of a penetrating agent and a dispersant is strong when penetrating agents exist mostly in ink, in the case of the pigment in which self-distribution is possible, there are such no worries in water. Moreover, since the particle diameter of a pigment is large and it cannot pass the opening in a recorded material easily compared with a color even if it uses the same penetrating agent about a drying property and a quality of printed character, too much osmosis is suppressed, and even if the drying time is early, it is thought that there are few blots. Moreover, surface tension becomes 40 or less mN/m, and scratch-proof nature is considered that wetting is good, the touch area of ink and a recorded agent spreads and fixing nature is improving by the bird clapper, although wetting to a recorded agent is bad in the case of the ink of high surface tension. Furthermore, since there is no bad influence by the interaction of a dispersant and a penetrating agent about blinding compared with usual pigment-content powder ink when the rate of a penetrating agent increases on a nozzle front face, it is thought that it becomes advantageous.

## EXAMPLE

[Example] Hereafter, an example explains this invention in more detail.
(Example 1) Microjet Black After diluting CW-1 (Orient company make) with water so that carbon concentration may become 10wt(s)%, centrifugal separation processing (7000r.p.m., 30 minutes) was carried out, and pigment dispersion liquid (carbon concentration 8.3wt%) were obtained.
The above-mentioned pigment dispersion liquid 50 weight sections glycerol (S. P. value : about 20) 15 weight sections diethylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=34 mN/m) 5 weight sections N, N-screw (2-hydroxyethyl)-2-aminomethyl ethane sulfonic acid 0.5 weight section NaOH 0.1 weight sections pure water Each component of the 29.4 weight sections above. It mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared.
[0039] (Example 2) Centrifugal separation processing (8000r.p.m., 40 minutes) of Cab-o-jet -300 (Cabot Corp. make) was carried out, and pigment dispersion liquid (carbon concentration 14.4wt%) were obtained.
The above-mentioned pigment dispersion liquid 35 weight sections ethylene glycol (S. P. value : about 18) 10 weight sections propylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=26 mN/m) 7 weight sections urea 5 weight sections pure water Each component of 43 weight sections above. It mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared.
[0040] (Example 1 of comparison) Not using the diethylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=34 mN/m), ink was prepared like the example 1 except having made the amount of pure water into the 34.4 weight sections. In addition, the S.P. value of a glycerol is 20 and is gamma=63 mN/m.
[0041] (Example 2 of comparison) Ink was prepared like the example 2 except having made the amount of the propylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=26 mN/m) into the 0.5 weight section, and having made the amount of pure water into 50 weight sections.
[0042]
(Example 3 of comparison)
Carbon black (Black Pearl L) 5 weight sections styrene-maleic-acid Na copolymer 1 weight section glycerol (S. P. value : about 20) 15 weight sections diethylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=34 mN/m) 5 weight sections N, N-screw (2-hydroxyethyl)-2-aminomethyl ethane sulfonic acid The 0.5 weight section NaOH 0.1 weight sections pure water A 73.4 weight sections carbon black and styrene-maleic-acid Na copolymer and purity. After preparing carbon black dispersion liquid, the component of above others was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared.
[0043]
(Example 4 of comparison)
Carbon black (Mitsubishi MA-100) A 4 weight sections styrene-maleic-acid Na copolymer 1 weight section glycerol (S. P. value : about 20, gamma=63 mN/m) 15 weight sections N, N-screw (2-hydroxyethyl)-2-aminomethyl ethane sulfonic acid The 0.5 weight section NaOH 0.1 weight sections pure water A 79.4 weight sections carbon black and styrene-maleic-acid Na copolymer

and pure water. After preparing carbon black dispersion liquid, the component of above others was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared.
[0044] (Example 1 of an examination) Following the (1) - (11) was evaluated using the ink obtained in examples 1 and 2 and the examples 1-4 of comparison.
(1) In the ink surface tension of 23 degrees C, and the environment of 55%RH, it measured using the UIRU helmet I type surface tension balance.
(2) In the ink viscosity of 23 degrees C, and the environment of 55%RH, it measured by shear-rate 1,400S-1.
(3) In the ink conductivity of 23 degrees C, and the environment of 55%RH, it measured using the conductivity meter (product made from AOL-40:DKK).
[0045] (4) Set at the diameter of ink number-average part granule application, and 23 degrees C of distributed particle size distributions, and it is Microtrup. UPA:Leeds & It measured using the product made from Northrup.
(5) Set at 23 degrees C of >0.5micrometer particle numbers contained in ink, and it is Accusizer. TM770 Optical Particle Sizer:Particle Sizing The acquired value was converted into the value per ink 1dm3 using the product made from Systems.
[0046] (6) 1 / 4tone (2035x128 pulse) was made to breathe out 3 times on the frequency of 6kHz using a trial production head (400dpi) in the amount of ink drops of 23 degrees C, and the environment of 55%RH, weight was measured in response to ink to the wafer of an ink absorber, and the regurgitation weight of 1 drops was found by calculation.
(7) Time after printing and printing the solid picture of 100% of 10mmx50mm rates of picture area in the paper using the thermal ink jet printer of resolution 600dpi made as an experiment to evaluation in drying-time test 23degree C and the environment of 55%RH, using FX-L paper (Fuji Xerox make) as a typical regular paper until a drop disappears from in the paper visually was measured.
[0047] (8) typical using the thermal ink jet printer of resolution 400dpi made as an experiment to picture quality test evaluation -- the printing test of 1dot line and a solid picture was usually performed in the paper, using FX-L paper (Fuji Xerox make) as paper As evaluation criteria, the homogeneity of a blot of a line and the edge of a solid picture was investigated, and evaluation was performed on the following criteria.
a) Line blot O [ ..... It is mustache-like b solid homogeneity with blot O in many portions. / ..... Disorder-less ** / ..... It is x with disorder slightly. / ..... Smoothness is missing by backlash backlash. ] ..... Blot-less ** ..... It spreads, is slightly and is x. [0048] (9) After printing the picture and passing after printing on the 1st using the thermal ink jet printer of resolution 400dpi made as an experiment to abrasion-resistance test evaluation, the picture section was ground against the finger and the following criteria estimated the dirt to the decoloring condition of the picture section, and the non-picture section.
O ..... [ ..... Picture concentration falls and the dirt of the non-picture section is conspicuous. ] The fall of picture concentration is not seen but the non-picture section is also completely dirt-less **..... Most picture concentration is x which has dirt in the non-picture section a little although it is not falling. [0049] (10) When it was left in 23 degrees C and the environment of 55% RH and **** resumption was carried out in the state where it does not act as a cap, after a **** halt using the thermal ink jet printer of resolution 400dpi made as an experiment to blinding-proof nature test evaluation, neglect time until picture disorder arises was measured. Evaluation was performed on the following criteria.
O ..... [ ..... Less than 0.5 minutes. ] More than 1 minute ** ..... 0.5 minutes - 1 minute x [0050] (11) After saving for one month in ink preservation stability test 60degree C and -20 degrees C, re-filtration with 1-micrometer filter was performed, and the ink after re-filtration was printed using the thermal ink jet printer of resolution 400dpi. Evaluation was performed on the following criteria.
O ..... With [ it is after preservation and / filterability ] no change. Picture-concentration-change-less ** ..... Although the fall of filtration velocity is seen a little [ after / preservation ], picture concentration is change-less x..... Filter plugging of the ink after preservation was intense, and

picture concentration fell greatly.
[0051]
[Table 1]
The table 1 ———— An example The example of comparison ———— 1 2 1 2 3 4 ———— Ink surface tension (mN/m) 37 34 6037 37 57 ———— Ink viscosity (mPas) 3.0 2.8 2.32.3 3.4 2.5 ———— Ink conductivity (S/m) 0.12 0.18 0.16 0.22 0.15 0.15 ———— Diameter of ink number-average part granule application (nm) 27 41 2540 3748 ———— A particle size distribution 1.8 2.2 1.8 2.02.7 2.3 ———— >0.5-micrometer particle number (x1010) 0.4 2.4 0.5 2.0 12.52.5 ———— The amount of ink drops (ng) 48 52 51 49 46 49 ———— drying-time test (second) 2 1 40 15 2 40 ———— Picture quality test a O O O x O O ———— Picture quality test bO O O O O ———— A wear-resistant test O Ox **O x ———— A blinding-proof nature test O O O O xO ———— Ink preservation stability test O O O O x O ———— [0052] (Example 3) Microjet Black 5-micrometer filter after carrying out centrifugal separation processing (8000r.p.m., 20 minutes) of CW-1 (Orient company make) -- subsequently pressure filtration was carried out with 2-micrometer filter, and pigment dispersion liquid (carbon concentration 15.7wt%) were obtained
The above-mentioned carbon black dispersion liquid . 25 weight sections triethylene-glycol monobutyl ether (S. P. value : about 10, gamma=34 mN/m) 4 weight sections propylene glycol (S. P. value : about 16) 10 weight sections SAFI Norian 440 (HLB 6 [ about ]) 0.3 weight sections urea 3 weight sections pure water The 57.7 weight sections. [0053] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 2.6mPas, 29 mN/m, 0.09 S/m, 24nm, 1.9, and 1.1x1010 pieces, respectively, and the amounts of drops were 51ng(s). All the results of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test of the drying time were O for 3 seconds.
[0054] (Example 4) After processing Cab-o-jet -300 (Cabot Corp. make) with an ultrasonic homogenizer for 30 minutes, centrifugal separation processing (7000r.p.m., 20 minutes) was diluted and carried out to 10% concentration with water, and pigment dispersion liquid (8.8% of carbon concentration) were obtained.
The above-mentioned carbon black dispersion liquid . 50 weight sections diethylene glycol (S. P. value : about 15) 10 weight sections N-methyl-2-pyrrolidone (S. P. value : about 11, gamma=48 mN/m) 5 weight sections dipropylene-glycol monobutyl ether (S. P. value : about 10, gamma=29 mN/m) 5 weight sections pure water 30 weight sections. [0055] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 2.5mPas, 33 mN/m, 0.18 S/m, 25nm, 2.1, and 1.6x1010 pieces, respectively, and the amounts of drops were 49ng(s). All the results of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test of the drying time were O for 1.5 seconds.
[0056] (Example 5) After carrying out scaling processing of the carbon black (Raven 5750) by the hypochlorous acid Na, it adjusted after [ pH / 7.5 ] desalting and distributed by using pure water as a solvent using the ultrasonic homogenizer. After distribution, centrifugal separation processing (7000r.p.m., 20 minutes) was carried out, and pigment dispersion liquid (11% of carbon concentration) were obtained.
The above-mentioned pigment dispersion liquid . 40 weight sections triethylene glycol (S. P. value : about 14) 10 weight sections propylene-glycol monopropyl ether (S. P. value : about 11,

gamma=26 mN/m) 8 weight sections polyoxyethylene alkyl ether 0.3 weight sections pure water The 41.7 weight sections. [0057] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 2.5mPas, 36 mN/m, 0.17 S/m, 59nm, 2.1, and 2.8x1010 pieces, respectively, and the amounts of drops were 47ng(s). For the result of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test, the drying time was O for 2 seconds.
[0058] (Example 6) After carrying out graft-polymer processing of styrene sulfonic-acid Na to carbon black (Mitsubishi MA-100), it distributed using the ultrasonic homogenizer by using pure water as a solvent, and after that, centrifugal separation processing (8000r.p.m., 40 minutes) was carried out, and pigment dispersion liquid (carbon concentration 7.8wt%) were obtained.
The above-mentioned pigment dispersion liquid . 60 weight sections thiodiethanol 20 weight sections polyoxyethylene polyoxypropylene blockpolymer 0.1 weight sections diethylene-glycol monochrome hexyl ether (S. P. value : about 10, gamma=29 mN/m) 3 weight sections pure water The 16.9 weight sections [0059] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 2.6mPas, 33 mN/m, 0.15 S/m, 47nm, 2.4, and 2.4x1010 pieces, respectively, and the amounts of drops were 53ng(s). All the results of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test of the drying time were O for 1 second.
[0060] (Example 7) After making it distribute by using water as a solvent using a high-pressure homogenizer so that carbon concentration may become 20% about the carbon black (Special Bk 4A) which performed plasma treatment, centrifugal separation processing (7000r.p.m., 30 minutes) was carried out, and pigment dispersion liquid (carbon concentration 15.6wt%) were obtained.
The above-mentioned pigment dispersion liquid 5 weight sections trimethylol propane . 15 weight sections glycerol (S. P. value : about 20) 5 weight sections propylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=26 mN/m) 5 weight sections polyoxyethylene perfluoro alkyl ether (HLB 13 [ about ]) 0.01 weight sections pure water 70 weight sections. [0061] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 3.3mPas, 23 mN/m, 0.14 S/m, 44nm, 2.3, and 1.7x1010 pieces, respectively, and the amounts of drops were 44ng(s). All the results of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test of the drying time were O for 2 seconds.
[0062]
(Example 8)
Pigment dispersion liquid used in the example 2 . 30 weight sections 1,5-pentanediol (S. P. value : about 12.5) 10 weight sections dipropylene-glycol monoethyl ether (S. P. value : about 10.5, gamma=28 mN/m) 8 weight sections isopropyl alcohol 3 weight sections silicone polyoxyethylene addition product (HLB 7 [ about ]) 0.1 weight sections polyoxyethylene oleyl ether (HLB 12 [ about ]) 0.3 weight sections pure water The 48.6 weight sections. [0063] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 3.0mPas, 33 mN/m, 0.16 S/m, 40nm, 2.4, and 1.3x1010 pieces, respectively, and the amounts of drops were 31ng(s). Moreover, for the result of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test, the drying time was O for 4 seconds. In addition, the amount of drops measured discharge quantity on the same conditions as an example 1 using the trial production head of 600dpi.

[0064]
(Example 9)
Pigment dispersion liquid used in the example 1 . 50 weight sections dipropylene glycol (S. P. value : about 13) 15 weight sections diethylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=34 mN/m) 5 weight sections benzoic acid Na 0.5 weight sections styrene-acrylic-ester-acrylic-acid emulsion (average molecular weight 12000, acid number 20 [ about ]) 1.0 weight sections pure water The 28.5 weight sections. [0065] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The amounts of drops were 31ng(s) in the viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution and a >0.5micrometer particle number, 3.2mPas, 36 mN/m, 3.2mPas, 0.22 S/m, 40nm, 2.4, and 1.5x1010 pieces. Moreover, for the result of a picture quality test, an abrasion-resistance test, a blinding-proof nature test, and an ink preservation stability test, the drying time was O for 1.5 seconds. In addition, the amount of drops measured discharge quantity on the same conditions as an example 1 using the trial production head of 600dpi.
[0066]
(Example 10)
Pigment dispersion liquid used in the example 3 . 30 weight sections glucose 10 weight sections 1,5-pentanediol (S. P. value : about 12.5) 10 weight sections dipropylene-glycol monopropyl ether (S. P. value : about 10.5, gamma=28 mN/m) 12 weight sections SAFI Norian 104 0.1 weight sections N-(2-acetamide) iminodiacetic acid The 0.5 weight sections LiOH 0.1 weight sections pure water The 37.3 weight sections. [0067] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 3.4mPas, 30 mN/m, 0.22 S/m, 25nm, 2.1, and 3.9x1010 pieces, and the amounts of drops were 45ng(s). For the result of 1s, a picture quality test, an abrasion-resistance test, and a blinding-proof nature test, the drying time was O, and the result of an ink preservation stability test was **.
[0068] (Example 11) After making it distribute by using water as a solvent using a high-pressure homogenizer so that it may become 20% of pigment concentration about a plasma treatment phthalocyanine pigment, centrifugal separation processing (7000r.p.m., 30 minutes) was carried out, and activated-charcoal-absorption processing was performed, and it filtered with 5-micrometer filter after that, and pigment dispersion liquid (14.7% of phthalocyanine-pigment concentration) were obtained.
The above-mentioned pigment dispersion liquid . 25 weight sections glycerol (S. P. value : about 20) 15 weight sections dipropylene-glycol monobutyl ether (S. P. value : about 10, gamma=29 mN/m) 5 weight sections polyoxyethylene polyoxypropylene block copolymer (HLB 10 [ about ]) The 0.3 weight sections PROXEL GXL0.03 weight section pure water The 53.7 weight sections.
[0069] Each above-mentioned component was mixed enough, pressure filtration was carried out with 2-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 2.8mPas, 32 mN/m, 0.22 S/m, 87nm, 2.4, and 5.7x1010 pieces, and the amounts of drops were 46ng(s). For the result of 1.5 seconds, a picture quality test, an abrasion-resistance test, and a blinding-proof nature test, the drying time was O, and the result of an ink preservation stability test was **.
[0070] (Example 12) Ink was prepared by the same composition and the same method as an example 11 except not performing centrifugal separation processing and activated-charcoal-absorption down stream processing of an example 11. The viscosity of ink was [ 32 mM/m, conductivity, the diameter of number-average part granule application particle size distribution, and the >0.5micrometer particle number of 2.8mPas(es) and surface tension ] 0.24 S/m, 110nm, 3.2, and 9.0x1010 pieces, respectively, and 15 ppm, 10 ppm, and the amounts of drops of the content of Fe and Mg were 46ng(s), respectively. The drying time was 1.5 seconds and, for the result of O, an abrasion-resistance test, an ink preservation stability test, and a blinding-proof nature test, the result of a picture quality test was **.

[0071]
(Example 5 of comparison)
Pigment dispersion liquid used in the example 1 . 50 weight sections diethylene glycol 15 weight sections tetraethylene-glycol monomethyl ether (S. P. value : about 10.5, gamma=50 mN/m) 5 weight sections thiourea 5 weight sections pure water 25 weight sections. [0072] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink was [ 47 mN/m, conductivity, the diameter of number-average part granule application particle size distribution, and the >0.5micrometer particle number of 2.7mPas(es) and surface tension ] 0.14 S/m, 28nm, 1.9, and 0.8x1010 pieces, respectively, and the amounts of drops were 53ng(s). Although the result of a picture quality test, a blinding-proof nature test, and an ink preservation stability test was O, for the result of an abrasion-resistance test, the drying time was x for 30 seconds.
[0073]
(Example 6 of comparison)
Pigment dispersion liquid used in the example 2 35 weight sections diethylene glycol 5 weight sections propylene-glycol monobutyl ether (S. P. value : about 10.5, gamma=26 mN/m) 20 weight sections pure water Each component of 40 weight sections above. It mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 3.2mPas, 33 mN/m, 0.16 S/m, 44nm, 2.8, and 7.5x1010 pieces, respectively, and the amounts of drops were 48ng(s). Although the result of the drying time of an abrasion-resistance test and a blinding-proof nature test was O for 1 second, the result of a picture quality test and an ink preservation stability test was x.
[0074]
(Example 13)
Pigment dispersion liquid used in the example 4 50 weight sections 1 and 2, 6-hexane triol (S. P. value : about 15) 10 weight sections ethylene glycol monopropyl ether (S. P. value : about 11, gamma=32 mN/m) 10 weight sections oxyethylene oleyl ether (HLB 10 [ about ]) 0.2 weight sections pure water The 29.8 weight sections. [0075] Each above-mentioned component was mixed enough, pressure filtration was carried out with 1-micrometer filter, and ink was prepared. The viscosity of ink, surface tension, conductivity, the diameter of number-average part granule application, particle size distribution, and the >0.5micrometer particle number were 2.5mPas, 39 mN/m, 0.13 S/m, 20nm, 2.1, and 1.2x1010 pieces, and the amounts of drops were 27ng(s). In addition, the amount of drops measured discharge quantity, in addition the amount of drops on the same conditions as an example 1 using the trial production head of 600dpi. A picture quality test, the abrasion-resistance test, the blinding-proof nature test, the drying-time test, and the ink preservation stability test were carried out using the thermal ink jet printer of resolution 600dpi which adopted the method of forming one drop by impressing the driving signal which consists of idle periods between the main pulse made as an experiment to evaluation, a pre pulse and a pre pulse, and a main pulse. All the other test results of the drying time were O in 4 seconds.
[0076] (Example 14) The ink of the following composition was prepared.
- Cyanogen ink Projet Fast Product made from Cyan.2:ZENECA 4 weight sections butyl carbitol 5 weight sections thiodiethanol 15 weight sections pure water Each component of 76 weight sections above. The mixed dissolution was carried out enough, pressure filtration was carried out with 0.45-micrometer filter, and ink was prepared.
[0077] - Magenta ink Projet Fast Instead of made from Cyan.2:ZENECA, it is Projet. Fast Magenta Except having used 2, the mixed dissolution of each component was enough carried out by the same composition, pressure filtration was carried out with 0.45-micrometer filter, and ink was prepared.
- Yellow ink Projet Fast Instead of made from Cyan.2:ZENECA, it is Projet. Fast Yellow Except having used 2, the mixed dissolution of each component was enough carried out by the same composition, pressure filtration was carried out with 0.45-micrometer filter, and ink was prepared.

[0078] (Example 2 of an examination)
(12) The following evaluation was carried out using the ink of the heavy picture quality test example 1 and the cyano ink obtained in the example 14, Magenta ink, and yellow ink.
[0079] The printing test of the solid picture pattern with which a black 1dot line [ as opposed to / usually using FX-L paper (Fuji Xerox make) as paper / a color background to in the paper / the ] and typical each color adjoin was performed using the thermal ink jet printer of resolution 400dpi made as an experiment to evaluation. As evaluation criteria, a blot of a line and the homogeneity of the solid picture contiguity section were investigated, and evaluation was performed on the following criteria.
[0080] a) Line blot O [ ..... It is mustache-like b solid homogeneity with blot O in many portions. / ..... Disorder-less ** / ..... It is x with disorder slightly. / ..... If smoothness was missing at all by backlash backlash, the evaluation result of a and b was O also about which ink. ] ..... Blot-less ** ..... It spreads, is slightly and is x.
[0081] (Example 3 of an examination) The following evaluation was carried out using the ink of an example 1, and the binder of the following composition.
- A binder A styrene-maleic-acid Li copolymer 5 weight sections ethylene glycol 15 weight sections polyoxyethylene oleyl ether 0.2 weight section isopropyl alcohol 3 weight sections pure water The thermal ink jet printer of resolution 400dpi made as an experiment to 76.8 weight sections evaluations is used, and it is a typical regular paper. Using FX-L paper (Fuji Xerox make), Binder A was piled up in the paper, the ink of an example 1 was piled up within 5 seconds after ****, it was made to print and the picture quality test of the above (9) was carried out. The result of a picture quality test is O and showed picture concentration higher than an example 1.
[0082] (Example 4 of an examination) The following evaluation was carried out using the ink of an example 2, and the binder of the following composition.
- Binder B benzalkonium chloride 1 weight section poly allylamine hydrochloride 5 weight sections glycerol 15 weight sections pure water The same picture quality test as the example 3 of 79 weight sections examinations was carried out. The result of a picture quality test is O and showed picture concentration higher than an example 2.
[0083] (Example 5 of an examination) The following evaluation was carried out using the ink of an example 1. The discharge quantity per 1 drops and the drying time were found like (6) of the example 1 of an examination, and (7) except having used the ink jet printer of 800dpi made as an experiment to evaluation. Moreover, the character of eight points was printed on FX-L paper (Fuji Xerox make) as a typical regular paper using the same printer. It excelled compared with the time of printing by the printer which 1 second and character grace used the discharge quantity per 1 drops by 13ng(s), and used the drying time by (8) of the example 1 of an examination.

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[Translation done.]